



LABNOTES

Summer 2000

The newsletter of the Wisconsin Laboratory Certification and Registration Program
Contact us: (608) 267-7633, LabCert@dnr.state.wi.us (email) or www.dnr.state.wi.us/org/es/science/lc

Marathon City Wastewater Treatment and Kohler Chemical and Metallurgical Testing Laboratories Honored

Greg Pils, WDNR Laboratory Certification Program

The 1999 Registered Laboratory of the Year awards were presented to Marathon City Wastewater Treatment Laboratory and the Kohler Company Chemical and Metallurgical Testing Laboratory at the March meeting of the Natural Resources Board. The awards, first presented in 1996, are intended to recognize those laboratories that have developed exceptional systems for producing high-quality data.

MARATHON CITY WASTEWATER TREATMENT PLANT LABORATORY

The Marathon City Wastewater Treatment Plant Laboratory, recognized with the award for a Small Registered Facility, provides analytical support for the city's wastewater treatment plant. They analyze samples for biochemical oxygen demand (BOD), total volatile solids, and total suspended solids.



Larry Heindl and family pose with Marathon City's 1999 Laboratory of the Year Award.

DNR Audit Chemist Rick Mealy did not identify a single deficiency related to quality assurance during the laboratory's June 1999 on-site evaluation. Mealy also noted that he was very impressed that the laboratory had consistently and appropriately qualified the data recorded on the Plant's discharge monitoring reports (DMRs) when necessary. Failure to qualify data on the DMR is one of the most common deficiencies identified during audits of wastewater treatment laboratories. The laboratory also analyzes replicate samples at a greater frequency than required for both treated and untreated samples. Mealy was most impressed with the quality of the laboratory's documentation and the attention given to record maintenance, both of which are critical to the performance of all laboratories performing environmental testing.

(see 1999 Laboratory of the Year, page 2)

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LABNOTES

Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor.

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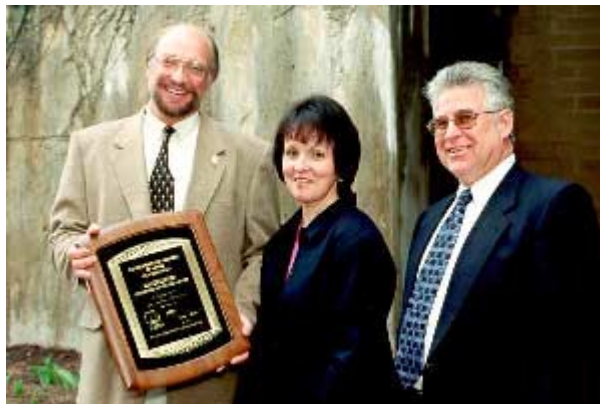
This document is available electronically at www.dnr.state.wi.us/org/es/science/lc.

This newsletter is intended to present current information and issues to certified and registered laboratories. This newsletter does not establish policy for the Department.

(1999 Laboratory of Year, Con't.)

KOHLER COMPANY CHEMICAL AND METALLURGICAL TESTING LABORATORY

The Kohler Company Chemical and Metallurgical Testing Laboratory was recognized with the award for a Large Registered Facility. The laboratory is registered for several fields of testing, including metals, volatile organic compounds and a full complement of inorganic analytes.



Lisa Escher (center) and Jerry Wilkinson (right) of Kohler Co. Chemical and Metallurgical Testing Laboratory with Jack Sullivan, WDNR Environmental Services Section Chief.

In his nomination, DNR Audit Chemist Greg Pils made special mention of the staff's commitment to produce data of the highest quality, and of how quickly the laboratory resolved the deficiencies identified during their June 1999 on-site evaluation. Pils also noted how the laboratory's staff works with the company's plant engineers to refine manufacturing processes and reduce amounts of pollutants in the company's waste stream. The laboratory also routinely employs techniques like the method of standard additions that, while more time consuming and labor-intensive, ultimately yield more accurate results.

NOMINATIONS FOR 2000 REGISTERED LABORATORY OF THE YEAR AWARDS

Nominations for the 2000 Registered Laboratory of the Year can be submitted by anyone – you don't have to be a DNR employee – are due December 31, 2000. To obtain a nomination form, contact Greg Pils by phone at (608) 267-9564, or via e-mail at pilsg@dnr.state.wi.us.

COUNCIL BIDS FAREWELL TO MANY AT MAY MEETING

The Laboratory Certification Standards Review Council held its quarterly meeting on May 18th and bid farewell to several members. Council Chair Ms. Mary Christie, Mr. Russ Janeshek, and Dr. Bill Sonzogni all are finishing their second term as representatives. Council members may serve up to two consecutive two-year terms. Council member Ms. Debbie Cawley has been reappointed to her second term and Mr. William Bruins has not sought reappointment. With these terms ending in July 2000 the next quarterly meeting will likely have several new representatives.

Current/pending appointments include representatives from the following areas: commercial laboratory sector, solid and hazardous waste, agricultural and State Laboratory of Hygiene. Council members are appointed by the Department of Administration; nominations may be forwarded through the Laboratory Certification Program or professional organizations. Information regarding the Council may be obtained by contacting Greg Pils, Department of Natural Resources at (608) 267-9564 or by e-mail: pilsg@dnr.state.wi.us.

CERTIFICATION STANDARDS REVIEW COUNCIL MEMBERS			
APPOINTMENT	REPRESENTATIVE	ADDRESS	PHONE, FAX & E-MAIL
Industrial Laboratory	David Kollakowsky	WI Electric Power Company P.O. Box 2046 Milwaukee, WI 53201	Phone: (414) 221-2835 Fax: (414) 221-4357 E-Mail: dave.kolloakowsky@wemail.wisenergy.com
Public Water Utility	Ruth Klee Marx	Marathon Cty. Health Dept. 1200 Lake View Dr. Rm 200 Wausau, WI 54403	Phone: (715) 882-7891 Fax: (715) 848-7160
Small Municipal WWTP	Gilbert Williams	Sun Prairie WPCF 300 East Main Sun Prairie, WI 53590	Phone: (608) 837-6292 Fax: (608) 835-6879
Large Municipal WWTP	Debbie Cawley	Green Bay Metro Sewer Dist. 2231 N. Quincy St. Green Bay, WI 54307	Phone: (920) 432-4893 Fax: (920) 432-4302
Agricultural Interest	VACANCY (Bruins)		
Commercial Laboratory	VACANCY (Christie)		
Demonstrated Interest	Marcia Kuehl	MAKuehl, Inc. 3470 Charlevoix Ct. Green Bay, WI 54311	
Solid and Hazardous Waste Disposal Facility	VACANCY (Hill)		
State Laboratory of Hygiene	George Bowman		

PROGRAM STAFF CHANGES

John R. "Jack" Sullivan, Chief of WDNR Environmental Science Services Section (which includes the Laboratory Certification and Registration Program), was appointed Director, of DNR's Bureau of Integrated Science Services on July 2, 2000.

Dan Olson, Program Assistant, left the Laboratory Certification Program earlier this spring to take a full-time position with DNR's Bureau of Community Financial Assistance.

Welcome to Summer Intern Nicole Hartz! Nicole recently received her BS in Biology/Environmental Science from UW-LaCrosse. Originally from Middleton, WI, Nicole is excited to be a part of the Lab Certification Program this summer. She will be working with the NELAC Implementation Team before beginning graduate studies at UW-Madison this fall pursuing a Master's degree in Environmental Monitoring.

CERTIFICATION AND REGISTRATION FEES FOR FY 2001

The Natural Resources Board approved the Laboratory Certification and Registration Program's FY 2001 fee schedule at their March 2000 meeting. The cost per relative value unit (RVU) increased from \$42.50 to \$47.00, resulting in total fee increases of \$63.00 for the typical wastewater treatment lab (registered lab base fee + categories 1-4) and \$266.00 for the typical commercial lab (certified lab base fee + categories 1-8, 10, 12, & 14-16). The increase is mainly due to the loss of RVUs since the start of FY 1999. For more information about how fees are determined, consult s. NR 149.05, Wis. Admin. Code, or contact Greg Pils at (608) 267-9564 or via e-mail at pilsg@dnr.state.wi.us.

Laboratory Fees for FY 2001 (Sept.1, 2000 - Aug. 30, 2001)

FEE ITEM	FY 2001 FEE	FEE ITEM	FY 2001 FEE
Registered Base Fee	\$470.00	Category 10	\$188.00
Certified Base Fee	\$705.00	Category 11	\$188.00
Reciprocity Fee	\$1,410.00	Category 12	\$188.00
Initial Application Fee	\$282.00	Category 13	\$188.00
Revised Application Fee	\$141.00	Category 14	\$188.00
Category 1	\$47.00	Category 15	\$564.00
Category 2	\$47.00	Category 16	\$188.00
Category 3	\$47.00	Category 17	\$564.00
Category 4	\$47.00	Category 18	\$940.00
Category 5	\$94.00	Category 18a (Nitrate Only)	\$94.00
Category 6	\$94.00	Category 18b (Nitrate & Fluoride)	\$188.00
Category 7	\$188.00	Category 19	\$188.00
Category 8	\$188.00	Category 20	\$1,222.00
Category 9	\$188.00	Category 21	\$188.00

WASTEWATER LAB FORUM

TO BOD OR CBOD, THAT IS THE QUESTION

Rick Mealy, Laboratory Certification Program

We are starting to see more and more wastewater treatment plants that undergo nitrification. Nitrification is the natural process where ammonia is first oxidized to nitrite and finally to nitrate. When nitrification occurs, BOD levels can elevate to the point of exceeding permit limits. If that's the case in your plant, it's probably time to consider a request that your permit specify testing and limits for carbonaceous BOD (CBOD) rather than conventional BOD.

The theoretical reaction of the oxidation of ammonia to nitrate requires approximately 4.57 milligram of oxygen for every milligram of ammonia oxidized. As with any biological reaction, temperature can increase and decrease its rate. Nitrification is inhibited at 10°C or less. This phenomenon may explain why you see changes in BOD to TSS ratios predominantly during the warmer months.

HOW DO YOU KNOW IF NITRIFICATION IS OCCURRING? You might be dealing with nitrification if any of the following apply to your system:

- effluent BOD is always significantly higher than TSS (e.g., TSS = 10, BOD = 25);
- effluent BOD is consistently higher than TSS in warmer months, but drops during winter months;
- your permit limit is 30 mg/L and you tend to run very close to that-- even though your plant runs well; or
- blank depletions are acceptable but GGA results are consistently biased high, and you use seed from your own process.

If you suspect nitrification is occurring, try running side by side BODs, inhibiting one set with TCMP (2-chloro-6-(trichloromethyl) pyridine) as specified in the sample pretreatment portion of Method 5210, *Standard Methods*.

If the inhibited BOD (CBOD) results are significantly lower and closer to TSS values, nitrification is occurring. If you recycle mixed liquor to the head of the plant, you could be adding nitrifying organisms to the influent. Some influents may contain nitrifiers even without recycle flows. In these situations, if you use your own plant as a seed source, it could mean high bias in your GGA data. Even facilities that totally nitrify may see some effect from conversion of ammonia in the dilution water.

WHAT CAN YOU DO IF NITRIFICATION IS OCCURRING? Switching your permit to CBOD may be the solution, but you must work with your DNR wastewater engineer before proceeding. The Department has established minimum-testing requirements that must be submitted before such a switch would be considered. Additional sampling for ammonia and/or nitrate or other sampling may also be required.

If you are successful in getting approval to test for CBOD rather than BOD, your facility's effluent limits will be modified to reflect that change.

SMOKING INTERFERES WITH LABORATORY TESTING

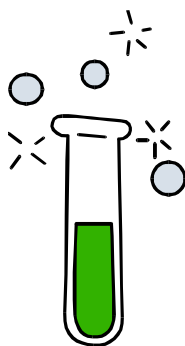
We're encountering many folks that either smoke in the laboratory or return to testing immediately after a "cigarette break". Smoking in the laboratory is, first and foremost, an issue of health and safety. But, did you know that cigarette smoke can lead to bias and contamination in analytical testing? Tobacco smoke and residue contains ammonia and contributes to BOD loading.

Perhaps of greater significance, are the phosphorus-laden residues on your fingers and hands. If you touch phosphorus glassware after smoking, the risk of contamination is considerable. If your blanks tend to show color, and you are a smoker, it is likely that smoking is the source of the contamination.

Being a non-smoker does not mean you are out of the woods. The oils in our skin are also a great source of phosphorus contamination. I ran into a lab

a few years back in which the analyst transferred all eight beakers at once to the hotplate by sticking his fingers inside the beakers. When the digestion was complete, and the analyst proceeded with the color reagent addition, ALL the samples, including the blank, were a deep blue color. Remember, contamination not only will result in the dreaded QC failures, but will result in high bias of results....and that may mean higher NR 101 fees!

The testing area must be smoke-free. If you do smoke, remember to wash your hands thoroughly with a non-phosphorus detergent afterwards. It's also a good idea to wear disposable laboratory gloves when performing the phosphorus test.



TRAINING UPDATE

One-day training programs on phosphorus and ammonia were held in locations throughout the state this spring. The sessions covered the electrode procedure for ammonia and the Test n' Tube and manual procedures for phosphorus.

If you were unable to attend previous training programs (Quality Assurance BOD, or Ammonia and Phosphorus), please contact Kay Marshall (*Wisconsin Rural Water*) at (715) 344-7778 or Rick Mealy at (608) 264-6006 or mealyr@dnr.state.wi.us. A list of interested parties will be developed and once enough interest is generated, encore presentations will be scheduled.

WHEN TO REPORT "LESS THAN" FOR BOD AND SUSPENDED SOLIDS

Tom Mugan, Bureau of Watershed Management

Many permittees ask how to report a BOD result when the dissolved oxygen depletion of the highest volume dilution is less than the minimum-required 2 mg/L. Should it be reported as "less than" something? How can I determine the limit of detection (LOD)? Can we get similar answers for suspended solids?

Recently, a group of Department technical staff investigated these and other questions, made a number of recommendations and wrote up their findings as guidance. This communication summarizes how the Department prefers that permittees analyze and report results when BOD and suspended solids levels are very low.

BOD

The BOD test method specifies that, for results to be reportable, the dissolved oxygen depletion must be at least 2 mg/L. It follows that the lowest reportable BOD result is 2 mg/L when straight sample (no dilution water) is tested. Because the minimum depletion is specified by the method and not determined in your lab as a method detection limit, we will call this number determined by the minimum depletion the **reporting limit** as opposed to calling it an LOD.

Does the test method require you to add nutrient-dosed dilution water to each bottle to provide the required nutrients? No. You can fill the BOD test bottle with straight sample and add small quantities of the nutrients directly to the BOD bottle. For convenience, at least one lab chemical supplier sells small packets of pre-measured nutrients, each packet suitable for one BOD test. Similarly, if you must add seed, small quantities of seed may be added directly to each BOD bottle, rather than using seeded dilution water.

SUSPENDED SOLIDS

The suspended solids test method instructs the analyst to optimize the solids loading on the filter by adjusting the volume of sample filtered. The Department suggests that we derive the reporting

limit for suspended solids from the 1-milligram minimum weight gain restriction from the *EPA Methods* manual. The Department believes that it is reasonable to increase sample size to at least 500 milliliters to obtain the 1-milligram minimum-required weight gain. In combination, these two restrictions allow reportable suspended solids results down to 2 mg/L or lower. We will call the number determined by the minimum residue restriction as the **reporting limit**.

SUMMARY OF GUIDELINES

- Permittees will not be expected to fill in the LOD or LOQ blanks on the Discharge Monitoring Reports for BOD and suspended solids.
- For BOD, if the oxygen depletion is less than 2 mg/L for all dilutions, report BOD as < (less than) the result that you would calculate for the highest volume dilution if the depletion were exactly 2 mg/L. If you used undiluted sample, report < 2 mg/L.
- If you frequently do not get the 2 mg/L minimum depletion, increase sample size of your highest volume dilution, up to undiluted sample, until you do.
- For suspended solids, if the residue is less than 1 milligram, report the suspended solids as < (less than) the result that you would calculate for the volume filtered if the residue were exactly 1 milligram. If you used 500 milliliters of sample, report < 2 mg/L.
- For the cleanest of samples, filter at least 500 milliliters. You may want to filter more.
- For averaging purposes on Discharge Monitoring Reports, assign a value of zero to any less-than result.
- Permittees using a commercial lab will need to provide additional sample volume so the lab can meet the desired reporting limits.

Department staff may contact permittees who frequently report "less than" values with a reporting limit greater than 2 mg/L to determine why sample size adjustments are not being made

You may find a copy of the full guidance document providing additional detailed explanations for these recommendations on the Laboratory Certification Program's web site. For questions or comments you

may contact Tom Mugan at (608) 266-7420 or via e-mail: mugant@dnr.state.wi.us.

REGULATORY UPDATE

The following *Federal Register* notices are of importance to the laboratory community:

Guidelines Establishing Test Procedures for the Analysis of Pollutants; Available Cyanide in Water; Final Rule (December 30, 1999). Promulgation of Method OIA-1677: Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry to Section 304(h) of the Clean Water Act.

National Primary and Secondary Drinking Water Regulations: Analytical Methods for Chemical and Microbiological Contaminants and Revisions to Laboratory Certification Requirements; Final Rule (December 1, 1999). Revised list of approved methods by updating revisions of ASTM, US EPA and *Standard Methods* analytical methods, as well as withdrew previous versions of 13 US EPA methods. Added new techniques for simultaneous determination of total coliforms and *E. coli*, 6 new methods for magnesium and 2 new methods for acid herbicides. Also included are changes to performance evaluation samples, requiring one successful PE per approved method per certification period.

Removal of the Maximum Contaminant Level Goal for Chloroform From the National Primary Drinking Water Regulations; Final Rule (May 30, 2000). Removes MCLG of zero for chloroform from SDWA regulation.

National Primary Drinking Water Regulations; Arsenic and Clarifications to Compliance and New Source Monitoring; Proposed Rule (June 22, 2000). Proposal to reduce arsenic MCLG to zero and MCL at 0.005 mg/L. Also seeking comment on MCLs of 0.003, 0.010 and 0.020 mg/L. Additionally includes proposals on monitoring after exceedances and new systems. **Comment period closes September 20, 2000.**

REFERENCE SAMPLE SUPPLIER APPROVAL

This fall, the Laboratory Certification Program will announce an updated list of reference sample suppliers. Applications were sent out in late June and will be reviewed after completion of renewal. Laboratories will be notified of the expanded list once approved by the Certification Standards Council.

NELAC

WISCONSIN NELAC LEGISLATION STALLED IN SENATE

Diane Drinkman, Laboratory Certification Program

After a promising unanimous vote in the State Assembly, AB 758, the proposed bill that would have authorized the DNR to apply for NELAP recognition, did not reach the Senate floor in time for a vote.

"We were very disappointed that AB 758 was allowed to languish without being heard in the Senate", said Alfredo Sotomayor, Senior Audit Chemist with the Laboratory Certification Program. "We had worked very hard to create these statute changes. Many thought that the unanimous approval by the Assembly was a good omen for passage by the Senate."

Since the legislature has adjourned for the rest of the year, and elections may change the profile of both houses, the Department will have to re-introduce the statutory changes next year. This will delay our application to NELAP and will extend timelines for offering NELAC accreditation in Wisconsin.

"I am still optimistic about the future of this proposed legislation. In the meantime, the Department's NELAC Implementation Team will continue to prepare the Certification program ready for a NELAP on-site sometime in the year 2001. We will publish a revised timeline on our web site", Sotomayor added.

LABORATORY SURVEY TO HELP DETERMINE WELAP

Dan Olson, Laboratory Certification Program

Earlier this year, laboratories that participate in the WDNR Laboratory Certification Program were surveyed to help determine the number of laboratories that will participate in the Wisconsin Environmental Laboratory Accreditation Program (WELAP).

A total of 429 labs that are active in the Laboratory Certification Program are located in Wisconsin. Some of these, 277 laboratories, were not surveyed because their WELAP accreditation status could be readily determined from their existing certification or registration status. Of those, 49 will require WELAP Accreditation (38 commercial and 11 public health laboratories); and the remainder (typically municipal registered laboratories) will not require WELAP Accreditation.

Of the 152 laboratories surveyed:

- 25 facilities will require WELAP Accreditation (2 Certified Municipal, 1 Certified Industrial, 3 Certified Hazardous Waste, 14 Registered Industrial and 5 Registered Hazardous Waste Laboratories);
- 7 facilities will voluntarily seek WELAP Accreditation (5 Municipal Certified and 2 Industrial Certified Laboratories); and
- 120 labs will not require or voluntarily seek WELAP Accreditation.

Combining data from all 429 Wisconsin labs:

- 74 labs will require WELAP Accreditation (17.3%);
- 7 labs will voluntarily seek WELAP Accreditation (1.6%); and
- 348 labs will not require or voluntarily seek WELAP Accreditation (81.1%).

AUDITOR'S CORNER

RAW LIKE SUSHI -- PART II

Alfredo Sotomayor, Senior Audit Chemist

In the first part of this article I argued how ambiguous definitions confused users by equating raw data with the media used to contain the data themselves. In this part I will focus on defining original observations, what I believe are the "real" raw data, and the need for retaining and having access to them.

Raw data are measured numbers. They are the most fundamental measurements needed to obtain, through a transforming function, a concentration or mass of analyte. Absorbance, emission counts, area counts, and peak heights are all examples of raw data. Once a number is transformed it no longer is raw. Usually, but not always, raw data are the numbers needed to be entered into a mathematical expression to quantify analytes. Thus, measured absorbance, raw data indeed, is used to obtain concentration of metals in atomic absorption analysis. However, calibration factors, although needed in some calibrations to obtain a concentration of analyte, are not raw because they are already transformed quantities, namely, the ratio of response to concentration of analyte and internal standard. If the fish is cooked, it is not sushi. Generally, the word "factor" precludes the associated quantity from qualifying as raw. So we could restate that raw data are the untransformed numbers necessary to obtain a concentration or mass of analyte.

Which may lead you to ask, how fundamental must the observation be? I am not advocating going down to the atomic level or to a primordial unmoved mover to substantiate a measurement. This is where the chosen calibration function can help us decide whether we are dealing with a sufficient level of "rawness". Absorbance is sufficiently raw to determine analyte concentrations in colorimetry and we need not measure light intensity by the photon to establish a valid relationship between measurement and analyte. It is the tissue that makes it sushi and

we need not consider the delicacy at the cellular level. However, once a transformation is effected, as when we compute a calibration factor, then we need to examine which measurements needed to be made to arrive at the factor. It is among those measurements that you will find the raw data.

THE BENTO BOX

Astoundingly, many analysts reared in the strictest empiricism (the "show me the money" school of chemistry), are nonetheless happy to abdicate knowledge of the transformations to which an instrument may submit raw data, or feel comfortable lacking access to sample raw data after a calibration event. And these days, when there is a proliferation of calibration algorithms and wide availability of sophisticated reduction software, it is more and more important to retain and to have access to original untransformed observations. Regulators and others need access to this information to:

- Verify that proper transformation and reduction techniques have been employed.
- Make sure that the best technique was used for a calibration.
- Monitor sudden anomalies in a signal.
- Enable future transformation, should other models become more appropriate, or if the original transformation is questioned.
- Submit information to alternative analyses.
- Divert questioning souls to the realm of higher mathematics.

The solution to this is to free yourself and those that you love from the tyranny of the black box, even when it is as attractive as a lacquered Japanese lunch box (bento), by insisting that any instrument you purchase give you access to all raw data.

ANALYTICAL INSTRUMENTS I HAVE KNOWN, OR WHAT'S IN THIS CALIFORNIA ROLL?

Instruments are still built that will not allow users access to raw data or that make this access incredibly convoluted. The main culprits are instruments used for inorganic analysis. There have been atomic absorption spectrophotometers that transformed calibration standard data by proprietary algorithms, disclosed virtually no information about the resulting calibration functions, and then only

provided concentrations of samples after a calibration event. There are inductively coupled plasma (ICP) emission spectrophotometers that provide no raw data after a calibration event. And some spectrophotometers are still sold by their manufacturer with preprogrammed calibration curves meant to be used in the "concentration" mode. For a contrast that will illustrate the point, consider how deprived an organic analyst would feel without peak areas.

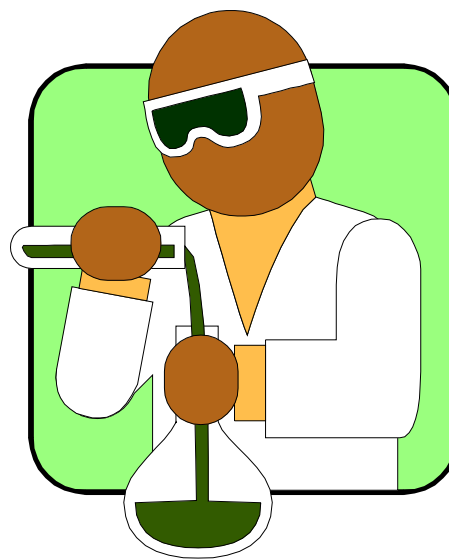
Recently, in the interest of quantitating low level concentrations as accurately as possible, I am seeing a proliferation of weighting techniques used in both organic and inorganic analyses. Whether this is desirable, justified, or appropriate could be the subject of another column. For now I will note that rarely do analysts get the full story on how their instruments weigh calibration data, and rarely can the manipulations to which the data is submitted be easily reproduced outside of the instrument. This then can become a raw data retention and traceability problem.

THE RECIPE

Since we all have to cook our data, regardless of how much we like them raw, try these basics. The ingredients are of course, good, unspoiled, and raw.

- **Retain raw data for all analyses.** This includes blanks, calibration standards, verification standards, and all samples analyzed. Make sure that all analytical instruments you use or purchase allow you access to all raw data. Some instruments, when operated in certain modes, do not always print raw data after a calibration event; however, if the data can be accessed on demand for any sample in any given run, and if this information is traceable to a printed concentration, this is quite acceptable.
- **Know the algorithm used to transform and reduce all calibration data.** Understand how all data is transformed in theory and in practice and be aware of the limitations of the algorithm chosen, if more than one is available. Test the accuracy or adequacy of the algorithm by running a few outside calculations.

- **Obtain a mathematical description of the calibration function.** This is your connection between raw data and concentrations. Even if you understand the algorithm used to generate calibrations, you must obtain and retain any equations, coefficients, or factors that uniquely describe each individual calibration.
- **Store all raw data in a format that ensures its permanence and accessibility.** Whether you keep raw data in hard copy or electronically, you must be able to save it and access it for the length of time required by regulations.
- **Ensure that you can reconstruct any transformation performed on any raw data.** You must be able to chronologically retrace any change to which raw data has been submitted. In the past I have written about reintegrating peaks and how you must always retain the original and subsequent areas. You must apply the same principle of chronological reconstruction to any raw data transformed to arrive at a concentration.
- **Avoid unnecessary data reduction and manipulations.** Processing for the sake of processing robs your data of its nutritional value and can invite more questions. Do not transform good raw data into "junk food". Try to base your transformations on established analytical laws and expected detector behavior. "Math magic" does not dazzle most regulators.



GO PHISH, OR HOW RAW IS PH?

I am afraid pH is neither fish nor fowl. It is decidedly not raw, and yet...Any pH reading converts the logarithm of the inverse of the concentration of hydrogen ion into a well-established scale. (Strictly speaking, pH electrodes measure the activity of the hydrogen ion.) Ions can be detected by measuring the electric potential of a solution, usually in millivolts. Does this mean then that I will expect to see millivolt readings for all pH measurements?

Most decidedly NOT. And this, rather than being a contradiction is explained by a universal law summarized by the Nernst equation. When one calibrates a pH meter one is really tuning the meter and not strictly calibrating it. Buffers are used to confirm that a pH electrode is operating as predicted by the Nernst equation (the equation's slope) and not to establish a different empirical relationship between pH and buffer concentration. By contrast, when one calibrates a GC or an AA, one establishes a relationship between analyte and response empirically, because that relationship is not governed by any universal pre-established quantitative law. Nevertheless, for pH meters and other detectors that operate according to the Nernst equation, monitoring millivolts can be useful to confirm a malfunction.

SUBSTANCES OF CONCERN AT LOW LEVELS

This list is published as a reminder that laboratories are required to report all data down to their limit of detection (LOD). All results greater than the LOD but less than the limit of quantitation (LOQ) must be reported and appropriately qualified (consult NR 149 for definitions of the LOD and LOQ). Be aware that some programs may require laboratories to report the results for *all* compounds to be reported down to the LOD, *even if they do not appear on this list*. Check with your clients to determine what reporting requirements apply. Labs may wish to institute the practice of always reporting all results down to the LOD, thereby avoiding confusion and insuring reporting requirements are always met.

Metals

Antimony
Beryllium
Cadmium
Lead
Thallium
Mercury
Chromium (Hexavalent)

Acids/Phenols

Pentachlorophenol (PCP)

Benzidines

Benzidine

Haloethers

Bis(chloromethyl)ether

Nitroaromatics

2,4-Dinitrotoluene
2,6-Dinitrotoluene

Polynuclear AromaticHydrocarbons

Benzo(a)pyrene

Phthalates & Adipates

Di(2-ethylhexyl)phthalate

Nonpurgeable ChlorinatedHydrocarbons

Hexachlorobenzene

Dioxins/Furans

Dioxin (2, 3, 7, 8-TCDD)

PCBs

Polychlorinated biphenyls

Chlorinated Pesticides

DDT and Metabolites
Heptachlor
Heptachlor epoxide
Lindane
Toxaphene

Carbamate Pesticides

Aldicarb

Nitrogen Pesticides

Alachlor
Dimethoate
Parathion
Trifluralin

Volatiles

1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,3-Dichloropropene (cis/trans)
Bromodichloromethane
Bromoform
Bromomethane
Chloroform
Chloromethane
Methyl tert-butyl ether (MTBE)
Methylene Chloride
Vinyl Chloride
Dibromochloropropane (DBCP)
Ethylene dibromide (EDB)



LABNOTES – SUMMER 2000

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